

PRECIPITATION IN Al-Mg₂Si ALLOYS

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Introduction

There exist several studies in the literature which are dealing with the mechanism of zone formation in Al-Mg₂Si alloys /1-8/. It is clear from these works /3-7/ that in the two-step ageing experiments the second ageing is largely influenced by the first even if the period of ageing at the first - generally the lower - temperature is very short. Nevertheless all the experiments, in which homogenized samples are quenched to room temperature have to be considered as two-step ageing experiments because in these cases the samples are kept unavoidably for some time at the quenching temperature. The effect of this unwanted annealing on the subsequent ageing process seems to be very important. The purpose of the present work is the study of the processes which can take place during the time between the quenching and ageing.

Experimentals

The alloys investigated contained 0.85, 1.42 and 1.75% Mg₂Si and the following impurities: Cu 0.04%, Mn 0.02 and Fe 0.016-0.035%. Wire samples 0.75 mm in diameter were homogenized at 555°C then quenched in room temperature water or in a -78°C brine.

The process of zone formation after quenching was followed by resistivity and dilatometric measurements. The resistivity measurements were carried out "in situ" at the temperature of ageing by continuous observation of the changes in resistivity. To diminish the interruption between the quenching and the beginning of the measurement the samples were mounted on the sample holder before homogenization, so the measurements could begin in less than 1 minute

after quenching. The period of the measurements was about 100 minutes, during this time the temperature of the samples was stabilized up to an accuracy about $5 \cdot 10^{-2}$ °C. Although according to previous investigations /5/ 3 or 4 successive quenching -and- ageing tests do not affect the reproducibility of the measurements, each sample was used only for one measurement.

To detect dimensional changes occurring during the ageing process a differential dilatometer of large sensitivity was used. The resolution of the dilatometer used in the investigations is 10^{-6} % relative length change. Variations were observed continuously on samples of 1.5 mm diameter and 10 mm in length. The period of the investigations was also about 100 minutes, one sample was used for 4 measurements. The uncontrolled period at room temperature between the quenching and the beginning of the measurement was about 2-3 min.

Results

a. Quenching to room temperature

Resistivity measurements were carried out on samples of each concentration between 30 and 200°C. The results obtained e.g. on the Al-1.75% Mg₂Si sample are shown in FIG.1. The main characteristics of the curves are as follows:

Resistivity was found to increase appreciably during ageing below 70°C. The process does not show any tendency for saturation, in agreement with previous results /5-8/. The rate of the resistivity change at the beginning of the process is increasing with the temperature. There is a gradual transition from a high to a low rate of reaction. The whole change in resistivity observed during the period of measurements becomes the smaller the higher is the temperature. Between 80 and 120°C the resistivity is still monotonously-increasing but a definite saturation trend can be observed. Decreasing resistivity appeared after relatively short periods of ageing between 140 and 200°C. Results of dilatometric measurements are shown in FIG.2.

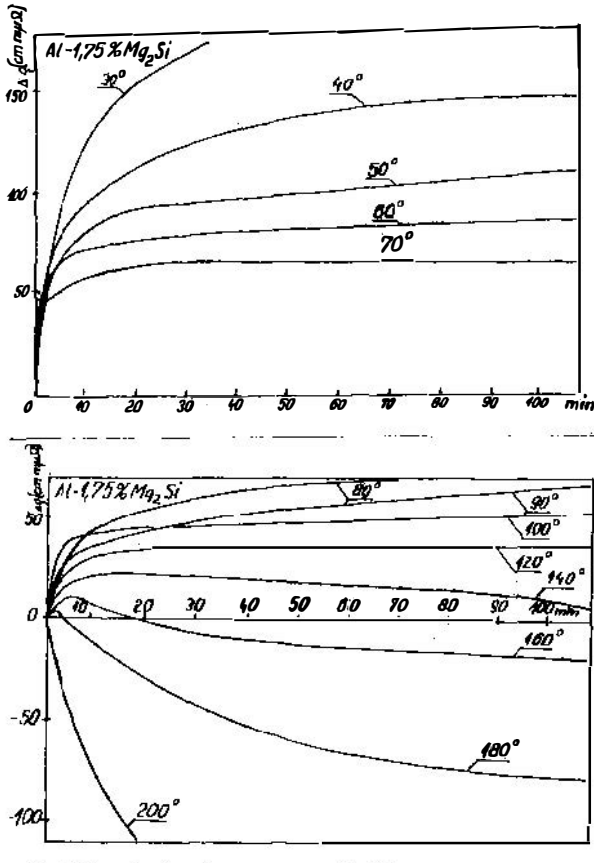


FIG.1.

Isothermal ageing curves

Because of the uncertainty of the periods between the quenching and the beginning of the measurements the curves of the length changes are less systematic than those of resistivity, their features, however, are entirely similar. The contraction in length in connection with zone formation seems to be an important observation.

The results described above show clearly that a small change in the temperature of ageing causes appreciable changes in the structure of the process, even near room temperature. So it is most expectable that by decreasing the tem-

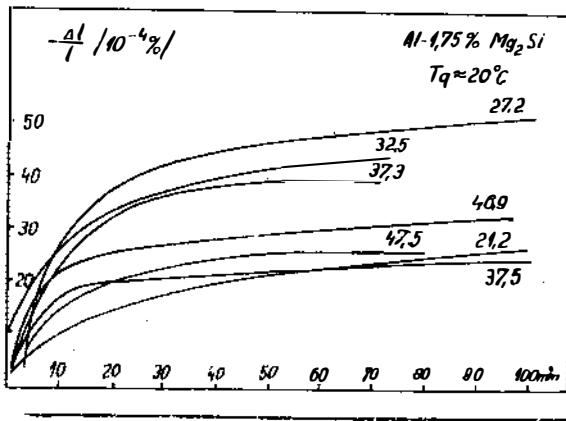


FIG. 2.

Results of dilatometric measurements

perature of ageing the early stages of the process can be slowed down and their features can better be observed. It is obvious too, that an appreciable part of the process is completed immediately after quenching to room temperature before the beginning of ageing. To avoid the quick completion of this process it is necessary to quench the samples to a lower temperature and to investigate the process of zone formation below the room temperature. In the following part we are to describe the results of measurements, which were carried out on samples quenched to -78°C .

b. Quenching to -78°C

To investigate the high rate reaction at the beginning of the ageing process resistivity measurements were made in the temperature range from -45°C to room temperature on samples quenched to -78°C . The results of these measurements are shown in FIG.3. The decrease in resistivity found at the first 15 min. of the ageing is considered to be the most important observation. The lower the temperature of ageing, the larger the decrease of resistivity. The maximum decrease in resistivity was about $30 \cdot 10^{-3} \text{ cm } \mu\Omega$. During the

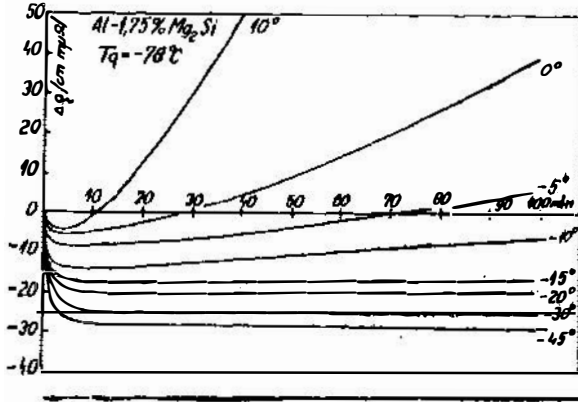


FIG.3.

Low temperature resistivity curves

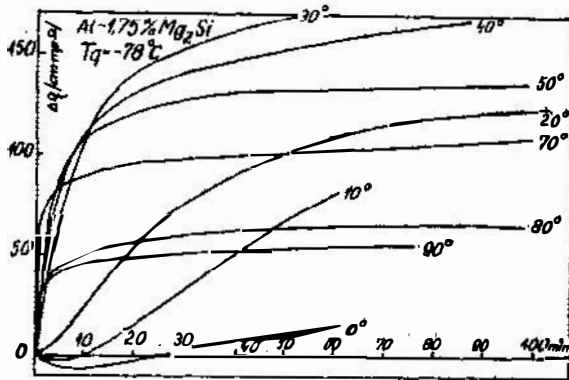


FIG.4.

Isothermal ageing curves after quenching to -78°C .

time investigated an increase in resistivity can only be observed at temperatures above -20°C . The increasing part of the curves at these temperatures starts the sooner, the higher the temperature is. If the temperature is raised further above 15°C no decrease is found any more. Above 30°C the curves are essentially the same as those after quenching to room temperature /FIG.4./ The maximum increase in resistivity upon ageing appears at about 30°C , the

magnitude of it is about $200 \cdot 10^{-3} \text{ cm } \mu\Omega$.

The dilatometric measurements carried out at room temperature or above it on samples quenched to -78°C do not differ appreciably from those made after quenching to room temperature.

In the time and temperature intervals of the investigations the alloys of different compositions have shown the same behaviour.

Summary

The ageing behaviour of Al-Mg₂Si alloys has been investigated by resistivity and length change measurements after quenching to room temperature and to -78°C . We have shown that the high rate process taking place at the beginning of the ageing near room temperature after both types of quenching must be a superposition of complicated part-processes. It appears that the models so far published are not able to account for these observations. That is why a correction of the models seems to be necessary.

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DISCUSSION :

- G.W. Lorimer : During the quench to -78°C in the alcohol/ CO_2 mixture a large number of vacancies will be lost due to the relatively slow rate of cooling ($2000^{\circ}\text{C}/\text{sec}$ for alcohol/ CO_2 as compared to $20000^{\circ}\text{C}/\text{sec}$ in H_2O). Also when the temperature falls below T_{GP} the amount of time available for the growth of G.P.zones is longer in the alcohol quench. The resulting distribution of G.P.zones and the rate of growth of the zones is a sensitive function of both the quenching rate and the quench temperature (P.N.T. G.W. Lorimer, R.S. Nicholson, Acta Met. 1969), and care must be taken when comparing samples which have undergone different quenching procedures. It would be interesting to compare samples quenched in alcohol to 0°C and -74°C . Also water quenched samples which were immediately cooled to -74°C would make interesting specimens.
- J. Lendvai : The loss of vacancies by quenching to -78°C alcohol/ CO_2 is found to be smaller than by quenching in water at room temperature, because the critical time available for vacancy migration is about 2 orders of magnitude larger when quenched in water, namely the sample has to be kept in the cooling medium for several seconds. We carried out some experiments on specimens quenched in alcohol at room temperature, and we compared the results with those obtained on samples quenched in water at room temperature. There was some slight difference in the actual rate of the process but the basic characteristic did not change at all.
- E. Nagy : The low quenching rate does not make any difference since there is a continuous transition from alcohol quench to the water quench. The process observed is most probably the binding of the vacancy to the impurity borne out by the activation energy for the resistivity drop (0.2 eV). The rise at -20° probably corresponds to the meeting of two such pairs, leading to the formation of G.P. zone nuclei, with $\Delta\rho > 0$.
- I. Kovacs : In connection with the question of Prof. Lorimer it must be emphasized that the quench to -78°C may not cause any loss in the properties. It is shown clearly by the fact that all the characteristics observed on samples quenched in water can be observed after alcohol quenching also if the ageing temperature is above room temperature. The alcohol quench makes therefore the ageing characteristics of this alloy surely richer and not poorer.